# Procedure for Solution Processing Polymers

## Chemicals:

* Ethanol
* Methanol
* Chlorobenzene
* P-xylene
* Toluene
* Polymers such as polyolefins, polythiophenes and polyfluorenes

## Methodology:

### Preparing the solution

* Clean appropriately sized glass vials, these may be 10, 20, or 4 mL in capacity depending on your intended solution volume
1. Remove the lids, which contain a foil-coated cardboard, and rinse the vials with hot, soapy water, then hot water
2. If the vials are to be used for applications with electrolytes, salt is a concern, so rinse the vial with deionized water at least 3 times
3. If the vials are to be used for general polymer solutions, rinse the vials once with ethanol from a solvent squirt bottle and pour the waste ethanol into an organics solvent waste container using a glass funnel (NB: always check that you are using the non-halogenated waste bottle for ethanol disposal, double check that ethanol is listed on the contents of the bottle)
4. Place the clean vials upside down onto the matte side of clean aluminium foil and place them into the glassware oven to dry overnight (NB: the matte side of the aluminium has not directly touched the rollers, whereas the shiny side is more likely to have come into contact with contaminants, like process grease.)
* Prepare a stock polymer solution. Using two stock solutions to make a blend is better than preparing blends using two solid components because it allows for greater concentration accuracy (by using large weights) and removes the complexity of accurately weighing two solids into the same vial. Always use mass rather than volume to measure a solution because density changes with temperature, particularly for solvents.
1. Determine the target concentration of the polymer solution, 1–1.4 wt% is common for solutions that will be spin coated or wire bar coated
2. Determine the desired volume, and calculate the amount of polymer needed
3. Clean a spatula and/or tweezers using toluene and a kimwipe, rinse the instruments with ethanol and dry with another kimwipe. Set the instruments on a clean piece of alumninium foil next to the balance.
4. Make sure that the clean, dry vial is at room temperature; heat will affect the readings on the mass balance
5. Measure the tare weight of the vial including the lid, record this value
6. Use the spatula or tweezers to approximately measure the appropriate amount of polymer into the vial
7. Ensure that the doors to the balance are closed before making a final measurement; remove the vial and allow the balance to rezero (but do not re-tare the balance!), then remeasure the mass. Record three measurements
8. Use a micropipetter to add in the approximately correct amount of solvent, weigh, and iterate until the desire mass is obtained. Weigh the solution 3 times.
9. For poor solvents, the polymer will not dissolve without heating. Place a water bath on the hot plate (use 80°C for toluene or p-xylene) with a magnetic stirbar at the bottom of the bath. Use a metal basket and clamp stand to suspend the vial in the water bath for 30 min.
* Prepare a stock solution of fullerene, dopant, or other additive using the same process as for the polymer
* Blend the stock polymer and additive solutions by pipetting the appropriate amounts into a clean vial. Work quickly to avoid solvent evaporation, and record all measurements on the basis of mass, not volume.

### Spin coating a thin film

* The substrate must be freshly cleaned to obtain high quality films
1. Cleave a silicon wafer into 10 x 10 mm pieces using a diamond tipped pen, take care not to touch the shiny side of the wafer with any instruments
2. Carefully holding the edges, rinse each substrate with toluene, ethanol, then DI water. Blow the substrate dry using a nitrogen gun.
3. Place each cleaned wafer, shiny side up, on the glass surface inside the UVO chamber. Run the UVO for 30 min. Use the samples immediately to avoid the adsorption of ambient species to the clean interface.
* Prepare the spincoater for use
1. Line the inside, bottom and top, with aluminium foil
2. Turn on the power point leading to the spin coater
3. Turn on the vacuum pump for the spin coater
4. Turn on the nitrogen supply
	1. Check that the regulator dial is all the way to the left, OFF
	2. Open the main cylinder valve until the cylinder pressure registers on the right side of the regulator
	3. Open the regulator valve until the left-hand gauge reads 4 bar, indicated by a black line on the gauge

Ensure that the t-junction valve is open to the spin coater

1. Select a programme and edit the duration and RPM if needed
2. Place a clean substrate onto the o-ring; ensure that the o-ring is entirely covered to avoid any solvent entering the pump
3. Press the vacuum button, the substrate should be held firmly onto the chuck
4. Dispense the target amount of solution onto the substrate, close the lid, and press go
5. Store spin coated samples in a glass or polystyrene petri dish using blue tack to hold them down, label each sample directly on the petri dish (not the lid of the petri dish)
6. Photograph the sample holder to prevent a future mixup
7. Clean the spincoater with a good solvent for the polymer used; for P3HT, use chlorobenzene. Be sure to remove the rubber o-rings before using any solvent harsher than ethanol. Cotton buds may help if the aperture is dirty.

### Safety Notes:

1. Wear covered clothing and safety glasses at all times when using equipment
2. Carefully observe methodology at all points
3. Use fume hood or similar apparatus when dealing with solvent vapors
4. Use nitrile gloves when dealing with hazardous materials/solutions
5. Use small volumes of solvent to minimize the effects of a spill
6. Transfer solvents by pipetting as much as possible to avoid the increased risks associated with pouring
7. Use the least toxic solvents possible in squirt bottles (e.g. tetrahydrofuran rather than toluene) to minimize the effects of unintended skin contact
8. Change gloves frequently when solvent splashes onto the nitrile (particularly important for tetrahydrofuran, which penetrates gloves and skin)
9. Perform all film processing, particularly the innovative techniques, in a fume cupboard
10. Clean glassware immediately after use to avoid more intense solvent exposure from trying to clean caked-on polymers
11. Use a funnel to collect the waste solvent from cleaning tweezers, spatulae, and stir bars
12. Store solvent waste in a secondary containment vessel away from sinks and drains
13. Take care to dispose of halogens like chlorobenzene, dichloromethane, or chloroform in the HALOGENATED waste container; other organic solvents like toluene, benzene, and ethanol must be placed in the NON-HALOGENATED waste container
14. Discuss modifications of the basic procedure with supervisor and update HazOp if required